Layered structures in proton-transfer compounds of 5-sulfosalicylic acid with the aromatic polyamines 2,6-diaminopyridine and 1,4-phenylenediamine

Graham Smith,[a]* Urs D. Wermuth[a] and Peter C. Healy[b]

[a]School of Physical and Chemical Sciences, Queensland University of Technology, GPO Box 2434, BRISBANE 4001, Australia, and [b]School of Science, Griffith University, NATHAN, 4111, Australia

Correspondence email: g.smith@qut.edu.au

The crystal structures of two proton-transfer compounds of 3-carboxy-4-hydroxybenzenesulfonic acid (5-sulfosalicylic acid) with the aromatic polyamines 2,6-diaminopyridine, $C_5H_8N^+ \cdot C_7H_5O_6S^- \cdot H_2O$, (I) and 1,4-phenylenediamine, $C_6H_{10}N_2^{2+} \cdot C_7H_4O_6S^{2-}$, (II) have been determined. Both compounds feature extensively hydrogen-bonded three-dimensional layered polymer structures having significant inter- layer π–π interactions between cation and anion species. With (I) the pyridine-N of the Lewis base is protonated and gives a direct hydrogen-bonding interaction with the water molecule which together with the two amine groups of the cation and the carboxylic acid group of the anion also give additional interactions with O-acceptors of the sulfonate group. With (II), a dianionic species results from deprotonation of both the sulfonic and carboxylic acid groups and all available O-acceptors interact with all dication donors.

Comment

The systematics of the solid-state structures of the proton-transfer compounds of 3-carboxy-4-hydroxybenzene-sulfonic acid (5-sulfosalicylic acid = 5-SSA) with Lewis bases have been widely studied because of the good crystallinity of many of the compounds. This feature is a result of the presence of sulfonate and potentially carboxyl oxygen acceptors available for hydrogen-bonding interactions. This has been useful for the study of certain difficult-to-crystallize bases, e.g. theophylline (a monohydrate) (Madarasz *et al.*, 2002), trimethoprim (a dihydrate) (Raj *et al.*, 2003) and pyrimethamine (a monohydrate) (Hemamalini *et al.*, 2005). However, examples of the dianionic 5-SSA species are rare, being found only in bis(guanidinium) 5-sulfosalicylate monohydrate (Smith, Wermuth & Healy, 2004). Although anhydrous compounds of 5-SSA are known, e.g. the 1:1 compounds with guanidine (Zhang *et al.*, 2004) and 1,10-phenanthroline (Fan *et al.*, 2005), the structures usually incorporate at least one water solvate molecule which acts in a donor/acceptor capacity, usually involving the aminium group in a direct hydrogen-bonding interaction. This, with additional interactions results in mostly three- dimensional polymer structures which in only a small number of the cases (those compounds with polycyclic heteroaromatic amines) involve π–π stacking effects (Smith, Wermuth & White, 2004).

Aniline-type proton-transfer compounds lend themselves to structure building since the protonated primary amine group will often give up to six interactions with available acceptor atoms. The structures of the 1:1 compounds of 5-SSA with aniline (Bakasova *et al.*, 1991), the 4-X-substituted anilines (X = F, Cl, Br) (Smith *et al.*, 2005a) and 4-aminobenzoic acid (Smith *et al.*, 2005b) have been reported. However, apart from the structures of two 5-SSA compounds with diamines [ethylenediaminium bis(5-sulfosalicylate) tetrahydrate (Gao *et al.*, 2004) and 4,4'-bipyridinium bis(5-sulfosalicylate) dihydrate] (Muthiah *et al.*, 2003), no polyfunctional aniline-

type compounds are known. We therefore attempted to obtain crystalline compounds of 5-SSA with aromatic polyamines with the aim of maximizing structure enhancement through both hydrogen bonding and possibly π–π interactive effects. The work has yielded limited success to date, but the two compounds whose crystal structures are reported here represent exceptions where good crystalline products were obtained. These compounds resulted from the reaction of 5-SSA with the aromatic polyamines 2,6-diaminopyridine (DAP) and 1,4-phenylenediamine (PDA), respectively 2,6-diaminopyridinium 5-sulfosalicylate monohydrate, $C_5H_8N^+ \cdot C_7H_5O_6S^- \cdot H_2O$, (I) and 1,4-phenylenediaminium 5-sulfosalicylate $C_6H_{10}N_2^{2+} \cdot C_7H_4O_6S^{2-}$, (II). Figs. 1 and 2 show the atom numbering schemes used for the 5-sulfosalicylate anion and aminium cations in (I) and (II) and are consistent with those previously employed in 5-SSA structures reported by this group (Smith, Wermuth & Healy, 2004; Smith, Wermuth & White, 2004; Smith *et al.*, 2005a, 2005b). Both (I) and (II) involve proton-transfer with subsequent extensive hydrogen bonding involving available proton-donor and acceptor atoms of both cation and anion species giving in both, three- dimensional layered polymer structures (Tables 1a and 1b).

With the 1:1 monohydrate DAP^+ 5-$SSA^- \cdot H_2O$, (I) (Fig. 3) single proton transfer to only the pyridine hetero nitrogen occurs and this group subsequently participates in a single hydrogen-bonding interaction with the water molecule [N—H···O, 2.775 (5) Å]. The water molecule also provides hydrogen-bonding links between sulfonate-O acceptors extending down the c-cell direction [O1W···.O53, 2.897 (5); O1W··· $O51^{i}$, 2.789 (5) Å], giving a total of five interactions for the sulfonate groups, including one with the carboxylic acid group of the 5-SSA anion [O71—H71···$O51^{ii}$, 2.621 (4) Å]. The result is the formation of an undulating layer structure in which the alternating DAP cations and 5-SSA anions partially superimpose down the c axial direction, with significant π–π ring interactions [ring centroid separation Cg···Cg = 3.54 (1) Å (intra); 3.56 (1) Å (inter)]. The overall result is a three-dimensional polymer structure. There is no occurrence of the $R^2_2(8)$ dimer interaction found in the small number of reported cocrystals of DAP [(1:1) proton-transfer compounds with 2-nitrobenzoic acid (Smith *et al.*, 1999) and 2,4,6-trinitrobenzoic acid (Smith *et al.*, 2000)]. However, this is probably because of the interjection in (I) of the water molecule into the hydrogen-bonding pattern.

The structure of the 1:1 compound of 5-SSA with 1,4-phenylenediamine, PDA^{2+} 5-SSA^{2-}, (II), except for the layering, differs markedly from that of (I) and those of the majority of the proton-transfer compounds of 5-SSA. The most unusual feature is the presence of dianionic 5-SSA species despite the use of 1:1 stoichiometric reactant ratios in the preparation. Both amine groups of the PDA molecule are protonated, which is also unusual considering that the second amine group is relatively acidic [$pK_{a1,2}$ 2.67, 6.60]. The crystallographic repeating unit comprises the 5-SSA^{2-} anion and two centrosymmetric PDA^{2+} half-cations (A and B) (Fig. 2). The A^{v} and B^{ix} molecular portions represent the inversion-generated halves of the two molecules [symmetry codes: (v) -x, -y, 1-z; (ix) 1-x, 1-y, -z]. The cation and anion molecules form into two-dimensional sheet structures through a number of hydrogen-bonding interactions involving all potential donor and acceptor atoms of both molecular species [range: 2.725 (3)–3.114 (2) Å]. These sheets are interlinked by N^+—H···O hydrogen-bonding interactions. (Fig. 4) and stack down the c cell direction with the alternating cation A··· anion··· cation B molecules separation indicating significant π–π interaction [inter-ring centroid distances: cation A···anion, 3.73 (1) Å; cation B···anion, 3.75 (1) Å]. The overall result is a three-dimensional polymer structure.

With the 5-SSA anion species in (I) and (II), similar structural and conformational features to those previously observed (Smith, Wermuth & Healy, 2004; Smith, Wermuth & White, 2004; Smith *et al.*, 2005a, 2005b) are found. The usual intramolecular hydrogen bond is found between the phenolic-OH group and a carboxyl oxygen (O2—H2···O72); for (I), 2.606 (4)Å, contracting as expected in (II) to 2.558 (2) Å where the carboxylic acid group is deprotonated. This also results in greater deviation from coplanarity of the overall group with the benzene ring [torsion angle C2–C1–C7–O71, 171.1 (2) ° (II) cf. 176.3 (3) ° (I)]. There is no occurrence of the strong intermolecular $R^2_2(8)$ cyclic carboxylic acid interaction as is found in the 4-chloro- and 4-bromoanilinium compounds with 5-SSA (Smith *et al.*, 2005a), nor of the anilinium-sulfonate $R^2_2(8)$ dimer interaction commonly found in the anhydrous guanidinium sulfonates (Russell *et al.*, 1994; Zhang *et al.*, 2004; Haynes *et al.*, 2004).

Experimental

The title compounds were synthesized by heating 1 mmol quantities of 3-carboxy-4-hydroxybenzenesulfonic acid (5-sulfosalicylic acid = 5-SSA) and respectively 2,6-diaminopyridine (DAP) or 1,4-phenylenediamine (PDA), in 50 mL of 50% ethanol-water for 10 min. under reflux. After concentration to ca. 30 mL, partial room-temperature evaporation of the hot-filtered solutions gave pale brown crystals of both (I) (m. p. 524–526 K) and (II) (m. p. >555 K).

(760gs23_SSADAP)

Crystal data

$C_7H_5O_6S·C_5H_8N_3·H_2O$	V = 723.9 (2) Å^3
M_r = 345.34	Z = 2
Monoclinic, $P2_1$	Mo $K\alpha$ radiation, λ = 0.71069 Å
a = 8.4778 (15) Å	μ = 0.27 mm^{-1}
b = 13.085 (2) Å	T = 297 K
c = 6.7562 (11) Å	0.40 × 0.30 × 0.30 mm
β = 105.001 (14)°	

Data collection

Rigaku AFC 7R diffractometer	R_{int} = 0.014
1965 measured reflections	3 standard reflections every 150 min
1726 independent reflections	intensity decay: 0.5%
1406 reflections with $I > 2\sigma(I)$	

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034	1 restraint
$wR(F^2)$ = 0.112	H atoms treated by a mixture of independent and constrained refinement
S = 0.89	$\Delta\rho_{max}$ = 0.25 e Å^{-3}
1726 reflections	$\Delta\rho_{min}$ = −0.24 e Å^{-3}
243 parameters	

Table 1

Selected geometric parameters (Å, º)

S5—O51	1.468 (3)	O72—C7	1.220 (5)
S5—O52	1.437 (4)	N11—C21	1.364 (5)
S5—O53	1.456 (3)	N11—C61	1.362 (5)
S5—C5	1.759 (4)	N21—C21	1.338 (6)
O2—C2	1.349 (5)	N61—C61	1.347 (5)
O71—C7	1.313 (5)		
O51—S5—O52	111.79 (19)	S5—C5—C4	119.3 (3)
O51—S5—O53	110.6 (2)	O71—C7—C1	113.8 (3)
O51—S5—C5	106.40 (18)	O72—C7—C1	122.7 (4)
O52—S5—O53	113.3 (2)	O71—C7—O72	123.4 (3)
O52—S5—C5	106.9 (2)	N21—C21—C31	124.2 (4)
O53—S5—C5	107.48 (18)	N11—C21—C31	118.3 (4)
C21—N11—C61	123.7 (4)	N11—C21—N21	117.4 (4)
O2—C2—C1	122.6 (3)	N11—C61—C51	118.1 (3)
O2—C2—C3	117.5 (3)	N11—C61—N61	117.5 (4)
S5—C5—C6	120.7 (3)	N61—C61—C51	124.4 (4)

Table 2

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O72	0.77 (8)	1.89 (8)	2.605 (4)	154 (8)
O1*W*—H1*A*···O51^{i}	0.79 (6)	2.02 (5)	2.789 (5)	163 (6)
O1*W*—H1*B*···O53	0.85 (6)	2.10 (5)	2.897 (5)	157 (6)
N11—H11···O1*W*	0.78 (4)	2.00 (4)	2.775 (5)	173 (4)
N21—H21*A*···O2^{ii}	0.76 (6)	2.35 (6)	3.054 (5)	154 (6)
N21—H21*B*···O52^{iii}	0.94 (5)	2.04 (5)	2.936 (6)	161 (4)
N61—H61*B*···O53^{iv}	0.81 (6)	2.49 (6)	3.252 (6)	158 (5)
O71—H71···O51^{iii}	0.93 (6)	1.71 (6)	2.621 (4)	166 (6)

Symmetry codes: (i) *x*, *y*, *z*+1; (ii) −*x*+1, *y*+1/2, −*z*+1; (iii) *x*−1, *y*, *z*; (iv) −*x*+2, *y*−1/2, −*z*+1.

(707gs09_SSAPDA)

Crystal data

$C_7H_4O_6S·C_6H_{10}N_2$	γ = 102.879 (14)°
M_r = 326.32	V = 668.4 (2) Å^3
Triclinic, $P\overline{1}$	Z = 2
a = 6.9800 (14) Å	Mo $K\alpha$ radiation, λ = 0.71069 Å
b = 9.1573 (15) Å	μ = 0.28 mm^{-1}
c = 10.849 (2) Å	T = 297 K
α = 84.618 (14)°	0.40 × 0.30 × 0.25 mm
β = 97.698 (16)°	

Data collection

Rigaku AFC 7R diffractometer	R_{int} = 0.034
3477 measured reflections	3 standard reflections every 150 min
3067 independent reflections	intensity decay: 0.8%
2678 reflections with $F^2 > 2\sigma(F^2)$	

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040	0 restraints
$wR(F^2)$ = 0.111	H atoms treated by a mixture of independent and constrained refinement
S = 0.83	$\Delta\rho_{max}$ = 0.28 e Å^{-3}
3067 reflections	$\Delta\rho_{min}$ = −0.30 e Å^{-3}
227 parameters	

Table 3

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O72	0.88 (4)	1.74 (4)	2.558 (2)	152 (4)
N1*A*—H11*A*···O71^{iii}	0.99 (4)	2.41 (4)	3.114 (2)	128 (3)
N1*A*—H11*A*···O72^{iii}	0.99 (4)	1.84 (4)	2.808 (3)	168 (3)
N1*B*—H11*B*···O53	0.96 (3)	1.90 (3)	2.812 (2)	157 (3)
N1*A*—H12*A*···O71^{iv}	0.91 (3)	1.83 (3)	2.725 (3)	166 (3)
N1*B*—H12*B*···O52^{v}	0.89 (3)	2.01 (4)	2.868 (3)	161 (3)
N1*A*—H13*A*···O2^{vi}	0.90 (3)	2.43 (3)	3.015 (3)	123 (2)
N1*A*—H13*A*···O51^{i}	0.90 (3)	2.14 (3)	2.905 (3)	143 (3)
N1*B*—H13*B*···O51^{vii}	0.84 (3)	1.95 (3)	2.794 (3)	177 (3)

Symmetry codes: (i) −*x*, −*y*, −*z*+1; (iii) −*x*+1, −*y*+1, −*z*+1; (iv) *x*−1, *y*, *z*; (v) −*x*+1, −*y*, −*z*; (vi) −*x*, −*y*+1, −*z*+1; (vii) −*x*, −*y*, −*z*.

Hydrogen atoms involved in hydrogen-bonding interactions (pyridinium, anilinium, carboxylic, phenolic and water) were located by difference methods and their positional and isotropic displacement parameters were refined. Others were included in the respective refinements at calculated positions (C—H = 0.93 Å) as riding atoms, with U_{eq} values fixed at 1.2U_{eq}(C).

Data collection: MSC/AFC Diffractometer Control (Molecular Structure Corporation, 1999) for 760gs23_SSADAP; *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1999) for 707gs09_SSAPDA. Cell refinement: MSC/AFC Diffractometer Control for 760gs23_SSADAP; *MSC/AFC Diffractometer Control Software* for 707gs09_SSAPDA. For both compounds, data reduction: *TEXSAN* for Windows (Molecular Structure Corporation, 1999); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON* (Spek, 2003).

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Figure 1

Fig. 1. Molecular configuration and atom-numbering scheme for the DAP^{+} cation, the 5-SSA^{-} anion and the water molecule in (I). Non-H atoms are shown as 30% probability displacement ellipsoids. Hydrogen atoms are shown small spheres of arbitrary size.

Figure 2

Fig. 2. Atom-numbering scheme for the two centrosymmetric half- PDA^{2+} cations (A and B) and the 5-SSA^{2-} anions in (II) (30% probability displacement ellipsoids). Symmetry codes: molecule A, (v) -x, -y, 1-z; molecule B, (ix) 1-x, 1-y, -z.

Figure 3

Fig. 3. Structure extension through hydrogen-bonding interactions (shown as broken lines) in (I), viewed in the unit cell down the a cell direction. Non-interactive H atoms are omitted for clarity. For symmetry codes see Table 2a.

Figure 4

Fig. 4. Perspective view of the hydrogen-bonding associations in the layered structure of (II) viewed in the unit cell down the a cell direction. Symmetry codes: (vii) x, y, 1+z; (viii) x, 1+y, z. For other symmetry codes, see Table 2b.

supplementary materials

Layered structures in proton-transfer compounds of 5-sulfosalicylic acid with the aromatic polyamines 2,6-diaminopyridine and 1,4-phenylenediamine

Graham Smith,* Urs D. Wermuth and Peter C. Healy

(2,6-diaminopyridinium 3-carboxy-4-hydroxybenzenesulfonate monohydrate')

Crystal data

$C_7H_5O_6S·C_5H_8N_3·H_2O$	$F(000) = 360$
$M_r = 345.34$	$D_x = 1.584$ Mg m^{-3}
Monoclinic, $P2_1$	Melting point: 524–526 K
Hall symbol: P 2yb	Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å
$a = 8.4778$ (15) Å	Cell parameters from 25 reflections
$b = 13.085$ (2) Å	$\theta = 12.6$–$15.8°$
$c = 6.7562$ (11) Å	$\mu = 0.27$ mm^{-1}
$\beta = 105.001$ (14)°	$T = 297$ K
$V = 723.9$ (2) Å^3	Prism, Pale brown
$Z = 2$	0.40 × 0.30 × 0.30 mm

Data collection

Rigaku AFC 7R diffractometer	$R_{int} = 0.014$
Radiation source: Rigaku rotating anode	$\theta_{max} = 27.5°$, $\theta_{min} = 2.9°$
graphite	$h = -4 \rightarrow 11$
ω–2θ scans	$k = 0 \rightarrow 17$
1965 measured reflections	$l = -8 \rightarrow 8$
1726 independent reflections	3 standard reflections every 150 min
1406 reflections with $I > 2\sigma(I)$	intensity decay: 0.5%

Refinement

Refinement on F^2	Primary atom site location: Structure-invariant direct methods
Least-squares matrix: Full	Secondary atom site location: Difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.034$	Hydrogen site location: Inferred from neighbouring sites
$wR(F^2) = 0.112$	H atoms treated by a mixture of independent and constrained refinement
$S = 0.89$	$w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 0.0953P]$ where $P = (F_o^2 + 2F_c^2)/3$
1726 reflections	$(\Delta/\sigma)_{max} = 0.002$
243 parameters	$\Delta\rho_{max} = 0.25$ e Å^{-3}
1 restraint	$\Delta\rho_{min} = -0.24$ e Å^{-3}

Special details

Geometry. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All esds are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S5	0.96025 (9)	0.09870 (7)	0.30700 (14)	0.0386 (3)
O2	0.4791 (4)	−0.2341 (2)	0.0940 (5)	0.0468 (9)
O51	1.0254 (3)	0.1158 (3)	0.1284 (5)	0.0524 (9)
O52	1.0837 (4)	0.0619 (3)	0.4803 (5)	0.0605 (10)
O53	0.8771 (4)	0.1898 (2)	0.3517 (6)	0.0559 (10)
O71	0.3248 (3)	0.0659 (2)	0.1281 (5)	0.0474 (9)
O72	0.2513 (3)	−0.0964 (2)	0.0559 (5)	0.0518 (9)
C1	0.5333 (4)	−0.0540 (3)	0.1445 (5)	0.0310 (9)
C2	0.5846 (4)	−0.1551 (3)	0.1372 (5)	0.0337 (10)
C3	0.7516 (5)	−0.1774 (3)	0.1769 (6)	0.0406 (11)
C4	0.8645 (4)	−0.0999 (3)	0.2260 (6)	0.0370 (10)
C5	0.8132 (4)	0.0014 (3)	0.2375 (5)	0.0323 (9)
C6	0.6490 (4)	0.0237 (3)	0.1961 (5)	0.0306 (9)
C7	0.3567 (4)	−0.0310 (3)	0.1056 (5)	0.0365 (10)
N11	0.6697 (4)	−0.0305 (3)	0.6979 (5)	0.0350 (9)
N21	0.4379 (5)	0.0680 (3)	0.6615 (6)	0.0478 (11)
N61	0.9169 (4)	−0.1142 (4)	0.7403 (7)	0.0511 (11)
C21	0.5036 (4)	−0.0244 (3)	0.6536 (5)	0.0333 (9)
C31	0.4141 (4)	−0.1134 (3)	0.6040 (6)	0.0382 (10)
C41	0.4959 (5)	−0.2037 (3)	0.6034 (6)	0.0410 (11)
C51	0.6640 (5)	−0.2084 (3)	0.6466 (6)	0.0406 (11)
C61	0.7528 (4)	−0.1194 (3)	0.6953 (5)	0.0367 (10)
O1W	0.8131 (4)	0.1616 (3)	0.7491 (6)	0.0576 (11)
H2	0.397 (9)	−0.208 (6)	0.092 (10)	0.10 (2)*
H3	0.786100	−0.246400	0.169100	0.0490*
H4	0.978200	−0.114700	0.252400	0.0440*
H6	0.611800	0.092100	0.204100	0.0370*
H71	0.214 (7)	0.076 (5)	0.111 (8)	0.067 (15)*
H11	0.714 (5)	0.022 (3)	0.706 (5)	0.023 (9)*
H21A	0.486 (7)	0.114 (5)	0.713 (8)	0.061 (17)*
H21B	0.324 (6)	0.070 (4)	0.634 (6)	0.039 (11)*
H31	0.298400	−0.112000	0.568900	0.0450*
H41	0.434500	−0.265300	0.570700	0.0480*
H51	0.719000	−0.271800	0.644600	0.0500*
H61A	0.941 (7)	−0.060 (5)	0.777 (8)	0.054 (16)*
H61B	0.965 (7)	−0.168 (5)	0.749 (8)	0.061 (17)*
H1A	0.887 (7)	0.155 (5)	0.847 (8)	0.063 (16)*
H1B	0.833 (7)	0.188 (5)	0.643 (8)	0.073 (17)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S5	0.0214 (4)	0.0424 (4)	0.0510 (5)	−0.0041 (4)	0.0074 (3)	0.0013 (4)
O2	0.0388 (14)	0.0384 (14)	0.0614 (17)	−0.0052 (12)	0.0095 (13)	0.0025 (13)
O51	0.0268 (11)	0.069 (2)	0.0646 (16)	−0.0042 (13)	0.0176 (11)	0.0144 (16)
O52	0.0386 (14)	0.068 (2)	0.0625 (17)	−0.0117 (15)	−0.0090 (13)	0.0044 (16)
O53	0.0382 (15)	0.0416 (16)	0.091 (2)	−0.0067 (13)	0.0225 (15)	−0.0119 (16)
O71	0.0245 (13)	0.0460 (15)	0.0730 (18)	0.0001 (11)	0.0149 (12)	−0.0074 (13)
O72	0.0279 (12)	0.0506 (17)	0.0720 (19)	−0.0086 (12)	0.0042 (12)	−0.0076 (15)
C1	0.0254 (15)	0.0369 (17)	0.0301 (15)	−0.0005 (12)	0.0063 (12)	0.0023 (13)
C2	0.0306 (16)	0.0359 (18)	0.0352 (16)	−0.0058 (13)	0.0095 (13)	0.0044 (14)
C3	0.0371 (18)	0.0338 (17)	0.052 (2)	0.0070 (15)	0.0134 (16)	0.0003 (15)
C4	0.0247 (15)	0.0411 (19)	0.0445 (19)	0.0030 (14)	0.0077 (13)	0.0016 (16)
C5	0.0254 (14)	0.0411 (18)	0.0307 (15)	−0.0049 (13)	0.0076 (12)	0.0027 (13)
C6	0.0250 (14)	0.0342 (16)	0.0340 (15)	−0.0020 (12)	0.0099 (12)	−0.0020 (13)
C7	0.0253 (15)	0.046 (2)	0.0377 (17)	−0.0046 (14)	0.0075 (13)	−0.0030 (15)
N11	0.0328 (15)	0.0372 (17)	0.0330 (14)	−0.0078 (13)	0.0048 (11)	0.0020 (12)
N21	0.040 (2)	0.0431 (19)	0.0548 (19)	0.0052 (15)	0.0024 (15)	−0.0035 (16)
N61	0.0326 (17)	0.056 (2)	0.061 (2)	−0.0022 (18)	0.0057 (15)	0.001 (2)
C21	0.0300 (15)	0.0398 (18)	0.0284 (15)	0.0017 (14)	0.0044 (12)	0.0013 (14)
C31	0.0298 (16)	0.045 (2)	0.0377 (18)	−0.0046 (15)	0.0051 (13)	0.0012 (15)
C41	0.0429 (19)	0.0388 (19)	0.0390 (18)	−0.0093 (16)	0.0064 (15)	−0.0017 (15)
C51	0.043 (2)	0.0352 (18)	0.0441 (19)	0.0048 (15)	0.0123 (16)	0.0027 (15)
C61	0.0309 (17)	0.045 (2)	0.0335 (17)	−0.0020 (15)	0.0072 (14)	−0.0006 (14)
O1W	0.0498 (18)	0.059 (2)	0.060 (2)	−0.0101 (17)	0.0069 (16)	−0.0014 (17)

Geometric parameters (Å, º)

S5—O51	1.468 (3)	N61—H61A	0.76 (6)
S5—O52	1.437 (4)	C1—C7	1.482 (5)
S5—O53	1.456 (3)	C1—C6	1.393 (5)
S5—C5	1.759 (4)	C1—C2	1.397 (5)
O2—C2	1.349 (5)	C2—C3	1.402 (6)
O71—C7	1.313 (5)	C3—C4	1.375 (6)
O72—C7	1.220 (5)	C4—C5	1.403 (6)
O2—H2	0.77 (8)	C5—C6	1.378 (5)
O71—H71	0.93 (6)	C3—H3	0.9547
O1W—H1A	0.79 (6)	C4—H4	0.9537
O1W—H1B	0.85 (6)	C6—H6	0.9551
N11—C21	1.364 (5)	C21—C31	1.383 (5)
N11—C61	1.362 (5)	C31—C41	1.371 (6)
N21—C21	1.338 (6)	C41—C51	1.380 (6)
N61—C61	1.347 (5)	C51—C61	1.379 (6)
N11—H11	0.78 (4)	C31—H31	0.9478
N21—H21A	0.76 (6)	C41—H41	0.9539
N21—H21B	0.94 (5)	C51—H51	0.9535
N61—H61B	0.81 (6)		

O51—S5—O52	111.79 (19)	C4—C5—C6	120.0 (3)

O51—S5—O53	110.6 (2)	S5—C5—C4	119.3 (3)
O51—S5—C5	106.40 (18)	C1—C6—C5	120.3 (4)
O52—S5—O53	113.3 (2)	O71—C7—C1	113.8 (3)
O52—S5—C5	106.9 (2)	O72—C7—C1	122.7 (4)
O53—S5—C5	107.48 (18)	O71—C7—O72	123.4 (3)
C2—O2—H2	102 (6)	C4—C3—H3	120.45
C7—O71—H71	111 (4)	C2—C3—H3	119.67
H1A—O1W—H1B	118 (6)	C3—C4—H4	120.15
C21—N11—C61	123.7 (4)	C5—C4—H4	119.61
C61—N11—H11	121 (3)	C5—C6—H6	121.19
C21—N11—H11	115 (3)	C1—C6—H6	118.47
H21A—N21—H21B	118 (6)	N21—C21—C31	124.2 (4)
C21—N21—H21A	124 (5)	N11—C21—C31	118.3 (4)
C21—N21—H21B	116 (3)	N11—C21—N21	117.4 (4)
H61A—N61—H61B	134 (6)	C21—C31—C41	118.7 (3)
C61—N61—H61A	107 (5)	C31—C41—C51	122.2 (4)
C61—N61—H61B	116 (4)	C41—C51—C61	118.9 (4)
C6—C1—C7	120.6 (3)	N11—C61—C51	118.1 (3)
C2—C1—C6	119.6 (3)	N11—C61—N61	117.5 (4)
C2—C1—C7	119.7 (3)	N61—C61—C51	124.4 (4)
O2—C2—C1	122.6 (3)	C41—C31—H31	120.50
O2—C2—C3	117.5 (3)	C21—C31—H31	120.78
C1—C2—C3	119.9 (3)	C31—C41—H41	118.88
C2—C3—C4	119.9 (4)	C51—C41—H41	118.89
C3—C4—C5	120.2 (3)	C41—C51—H51	121.18
S5—C5—C6	120.7 (3)	C61—C51—H51	119.95

O51—S5—C5—C4	74.2 (3)	C2—C1—C6—C5	0.7 (5)
O51—S5—C5—C6	−106.3 (3)	C7—C1—C6—C5	178.2 (3)
O52—S5—C5—C4	−45.4 (3)	C7—C1—C2—O2	0.6 (5)
O52—S5—C5—C6	134.1 (3)	O2—C2—C3—C4	−178.6 (3)
O53—S5—C5—C4	−167.4 (3)	C1—C2—C3—C4	1.0 (5)
O53—S5—C5—C6	12.1 (3)	C2—C3—C4—C5	0.3 (6)
C21—N11—C61—C51	−0.5 (5)	C3—C4—C5—S5	178.4 (3)
C61—N11—C21—N21	−179.8 (3)	C3—C4—C5—C6	−1.1 (5)
C61—N11—C21—C31	0.3 (5)	S5—C5—C6—C1	−178.9 (3)
C21—N11—C61—N61	179.4 (4)	C4—C5—C6—C1	0.6 (5)
C6—C1—C7—O72	178.4 (3)	N11—C21—C31—C41	0.6 (5)
C6—C1—C7—O71	−1.1 (5)	N21—C21—C31—C41	−179.3 (4)
C2—C1—C7—O72	−4.1 (5)	C21—C31—C41—C51	−1.3 (6)
C2—C1—C7—O71	176.4 (3)	C31—C41—C51—C61	1.1 (6)
C6—C1—C2—O2	178.1 (3)	C41—C51—C61—N61	180 (2)
C6—C1—C2—C3	−1.5 (5)	C41—C51—C61—N11	−0.2 (5)
C7—C1—C2—C3	−179.0 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O72	0.77 (8)	1.89 (8)	2.605 (4)	154 (8)
O1*W*—H1*A*···O51^{i}	0.79 (6)	2.02 (5)	2.789 (5)	163 (6)
O1*W*—H1*B*···O53	0.85 (6)	2.10 (5)	2.897 (5)	157 (6)
N11—H11···O1*W*	0.78 (4)	2.00 (4)	2.775 (5)	173 (4)

N21—H21*A*···O2^{ii}	0.76 (6)	2.35 (6)	3.054 (5)	154 (6)
N21—H21*B*···O52^{iii}	0.94 (5)	2.04 (5)	2.936 (6)	161 (4)
N61—H61*B*···O53^{iv}	0.81 (6)	2.49 (6)	3.252 (6)	158 (5)
O71—H71···O51^{iii}	0.93 (6)	1.71 (6)	2.621 (4)	166 (6)
C6—H6···O53	0.96	2.55	2.918 (5)	103
C6—H6···O71	0.96	2.38	2.723 (4)	101

Symmetry codes: (i) *x*, *y*, *z*+1; (ii) −*x*+1, *y*+1/2, −*z*+1; (iii) *x*−1, *y*, *z*; (iv) −*x*+2, *y*−1/2, −*z*+1.

(1,4-phenylenediaminium 3-carboxylato-4-hydroxybenzenesolfonate)

Crystal data

$C_7H_4O_6S·C_6H_{10}N_2$	$Z = 2$
$M_r = 326.32$	$F(000) = 340$
Triclinic, $P\overline{1}$	$D_x = 1.621$ Mg m^{-3}
Hall symbol: -P 1	Melting point: greater than 555 K K
$a = 6.9800$ (14) Å	Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å
$b = 9.1573$ (15) Å	Cell parameters from 25 reflections
$c = 10.849$ (2) Å	$\theta = 12.7$–$17.2°$
$\alpha = 84.618$ (14)°	$\mu = 0.28$ mm^{-1}
$\beta = 97.698$ (16)°	$T = 297$ K
$\gamma = 102.879$ (14)°	Prismatic, Pale brown
$V = 668.4$ (2) Å^3	0.40 × 0.30 × 0.25 mm

Data collection

Rigaku AFC 7R diffractometer	$R_{int} = 0.034$
Radiation source: Rigaku rotating anode	$\theta_{max} = 27.5°$, $\theta_{min} = 2.9°$
graphite	$h = -3 \rightarrow 9$
ω–2θ scans	$k = -11 \rightarrow 11$
3477 measured reflections	$l = -14 \rightarrow 13$
3067 independent reflections	3 standard reflections every 150 min
2678 reflections with $F^2 > 2\sigma(F^2)$	intensity decay: 0.8%

Refinement

Refinement on F^2	Primary atom site location: Structure-invariant direct methods
Least-squares matrix: Full	Secondary atom site location: Difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.040$	Hydrogen site location: Inferred from neighbouring sites
$wR(F^2) = 0.111$	H atoms treated by a mixture of independent and constrained refinement
$S = 0.83$	$w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 2.7752P]$ where $P = (F_o^2 + 2F_c^2)/3$
3067 reflections	$(\Delta/\sigma)_{max} = 0.002$
227 parameters	$\Delta\rho_{max} = 0.28$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.30$ e Å^{-3}

Special details

Geometry. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All esds are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S5	0.24133 (6)	−0.07178 (5)	0.17261 (4)	0.0220 (1)
O2	0.2200 (2)	0.53368 (17)	0.30520 (17)	0.0382 (5)
O51	0.0443 (2)	−0.16497 (16)	0.18292 (15)	0.0332 (4)
O52	0.3917 (2)	−0.12624 (16)	0.25917 (15)	0.0338 (4)
O53	0.2911 (2)	−0.05407 (17)	0.04508 (14)	0.0345 (5)
O71	0.7443 (2)	0.38757 (18)	0.38199 (16)	0.0382 (5)
O72	0.5953 (2)	0.58001 (17)	0.36193 (16)	0.0389 (5)
C1	0.4086 (3)	0.3451 (2)	0.29678 (17)	0.0214 (5)
C2	0.2298 (3)	0.3953 (2)	0.27685 (18)	0.0244 (5)
C3	0.0565 (3)	0.3015 (2)	0.2271 (2)	0.0288 (6)
C4	0.0594 (3)	0.1595 (2)	0.19590 (19)	0.0269 (5)
C5	0.2359 (3)	0.1086 (2)	0.21482 (17)	0.0220 (5)
C6	0.4085 (3)	0.2011 (2)	0.26493 (17)	0.0228 (5)
C7	0.5962 (3)	0.4427 (2)	0.35162 (18)	0.0256 (5)
N1A	0.0053 (3)	0.29317 (19)	0.56513 (18)	0.0282 (5)
C1A	0.0033 (3)	0.1417 (2)	0.53136 (17)	0.0232 (5)
C2A	0.1793 (3)	0.0934 (2)	0.54190 (19)	0.0263 (5)
C3A	0.1758 (3)	−0.0505 (2)	0.51007 (19)	0.0272 (5)
N1B	0.3612 (3)	0.21705 (19)	−0.10300 (18)	0.0269 (5)
C1B	0.4327 (3)	0.3632 (2)	−0.04953 (17)	0.0230 (5)
C2B	0.3014 (3)	0.4556 (2)	−0.0464 (2)	0.0289 (6)
C3B	0.3700 (3)	0.5945 (2)	0.0037 (2)	0.0296 (6)
H2	0.343 (6)	0.578 (4)	0.331 (4)	0.080 (12)*
H3	−0.064500	0.335800	0.213300	0.0340*
H4	−0.059900	0.095800	0.162100	0.0310*
H6	0.528700	0.166000	0.278300	0.0270*
H2A	0.301300	0.157800	0.570100	0.0300*
H3A	0.301100	−0.081600	0.518300	0.0210*
H11A	0.143 (5)	0.349 (4)	0.583 (3)	0.062 (9)*
H12A	−0.072 (4)	0.340 (3)	0.507 (3)	0.041 (7)*
H13A	−0.041 (5)	0.287 (3)	0.639 (3)	0.051 (8)*
H2B	0.165400	0.424400	−0.077900	0.0340*
H3B	0.288200	0.662200	0.017400	0.0210*
H11B	0.370 (5)	0.136 (4)	−0.041 (3)	0.054 (8)*
H12B	0.425 (5)	0.201 (4)	−0.165 (3)	0.063 (10)*
H13B	0.240 (4)	0.203 (3)	−0.130 (3)	0.041 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S5	0.0189 (2)	0.0168 (2)	0.0301 (3)	0.0022 (2)	0.0006 (2)	−0.0079 (2)
O2	0.0287 (8)	0.0241 (7)	0.0651 (10)	0.0088 (6)	0.0006 (7)	−0.0194 (7)
O51	0.0235 (7)	0.0235 (7)	0.0505 (9)	−0.0023 (5)	0.0033 (6)	−0.0110 (6)
O52	0.0327 (8)	0.0265 (7)	0.0434 (8)	0.0125 (6)	−0.0078 (6)	−0.0106 (6)
O53	0.0447 (9)	0.0274 (7)	0.0337 (8)	0.0066 (6)	0.0108 (6)	−0.0081 (6)
O71	0.0241 (7)	0.0354 (8)	0.0534 (10)	0.0065 (6)	−0.0104 (6)	−0.0156 (7)
O72	0.0304 (8)	0.0232 (7)	0.0616 (10)	0.0003 (6)	−0.0025 (7)	−0.0196 (7)
C1	0.0198 (8)	0.0188 (8)	0.0249 (9)	0.0018 (6)	0.0003 (6)	−0.0062 (6)
C2	0.0245 (9)	0.0194 (8)	0.0307 (9)	0.0061 (7)	0.0023 (7)	−0.0065 (7)
C3	0.0198 (9)	0.0272 (9)	0.0407 (11)	0.0082 (7)	−0.0019 (7)	−0.0085 (8)
C4	0.0195 (9)	0.0226 (9)	0.0369 (10)	0.0021 (7)	−0.0033 (7)	−0.0087 (7)
C5	0.0207 (8)	0.0177 (8)	0.0273 (9)	0.0029 (6)	0.0004 (7)	−0.0062 (6)
C6	0.0196 (8)	0.0202 (8)	0.0289 (9)	0.0048 (7)	−0.0004 (7)	−0.0064 (7)
C7	0.0234 (9)	0.0236 (9)	0.0283 (9)	0.0008 (7)	0.0004 (7)	−0.0087 (7)
N1A	0.0290 (9)	0.0190 (8)	0.0359 (9)	0.0044 (7)	−0.0023 (7)	−0.0092 (7)
C1A	0.0257 (9)	0.0171 (8)	0.0259 (9)	0.0031 (7)	−0.0002 (7)	−0.0053 (6)
C2A	0.0207 (9)	0.0229 (9)	0.0322 (10)	−0.0005 (7)	−0.0034 (7)	−0.0081 (7)
C3A	0.0205 (9)	0.0240 (9)	0.0365 (10)	0.0045 (7)	−0.0018 (7)	−0.0071 (7)
N1B	0.0235 (8)	0.0227 (8)	0.0346 (9)	0.0046 (6)	−0.0015 (7)	−0.0097 (7)
C1B	0.0246 (9)	0.0191 (8)	0.0251 (9)	0.0034 (7)	0.0021 (7)	−0.0046 (6)
C2B	0.0202 (9)	0.0278 (10)	0.0387 (11)	0.0053 (7)	−0.0020 (7)	−0.0094 (8)
C3B	0.0233 (9)	0.0266 (9)	0.0408 (11)	0.0087 (7)	−0.0007 (8)	−0.0098 (8)

Geometric parameters (Å, º)

S5—O51	1.4602 (15)	C1—C6	1.394 (3)
S5—O52	1.4538 (16)	C2—C3	1.393 (3)
S5—O53	1.4570 (16)	C3—C4	1.379 (3)
S5—C5	1.7646 (19)	C4—C5	1.396 (3)
O2—C2	1.350 (2)	C5—C6	1.386 (3)
O71—C7	1.242 (3)	C3—H3	0.9539
O72—C7	1.274 (2)	C4—H4	0.9539
O2—H2	0.88 (4)	C6—H6	0.9524
N1A—C1A	1.464 (3)	C1A—C3A^{i}	1.384 (3)
N1A—H13A	0.90 (3)	C1A—C2A	1.383 (3)
N1A—H12A	0.91 (3)	C2A—C3A	1.387 (3)
N1A—H11A	0.99 (4)	C2A—H2A	0.9522
N1B—C1B	1.465 (3)	C3A—H3A	0.9692
N1B—H12B	0.89 (3)	C1B—C2B	1.384 (3)
N1B—H11B	0.96 (3)	C1B—C3B^{ii}	1.382 (3)
N1B—H13B	0.84 (3)	C2B—C3B	1.390 (3)
C1—C2	1.407 (3)	C2B—H2B	0.9540
C1—C7	1.496 (3)	C3B—H3B	0.9645
O51—S5—O52	111.82 (9)	C4—C5—C6	119.87 (17)
O51—S5—O53	112.17 (9)	C1—C6—C5	120.84 (19)
O51—S5—C5	106.72 (9)	O72—C7—C1	117.39 (18)

O52—S5—O53	111.75 (9)	O71—C7—O72	123.64 (19)
O52—S5—C5	107.23 (9)	O71—C7—C1	118.95 (17)
O53—S5—C5	106.75 (9)	C2—C3—H3	120.10
C2—O2—H2	105 (2)	C4—C3—H3	119.64
H12A—N1A—H13A	109 (3)	C5—C4—H4	120.09
H11A—N1A—H12A	115 (3)	C3—C4—H4	119.79
C1A—N1A—H12A	111.1 (18)	C1—C6—H6	119.36
C1A—N1A—H13A	109.0 (17)	C5—C6—H6	119.80
C1A—N1A—H11A	110 (2)	N1A—C1A—C2A	119.39 (18)
H11A—N1A—H13A	103 (3)	N1A—C1A—C3A^{i}	118.80 (19)
H11B—N1B—H13B	104 (3)	C2A—C1A—C3A^{i}	121.82 (17)
C1B—N1B—H11B	111 (2)	C1A—C2A—C3A	118.96 (19)
H12B—N1B—H13B	107 (3)	C1A^{i}—C3A—C2A	119.22 (19)
C1B—N1B—H12B	115 (2)	C3A—C2A—H2A	120.31
C1B—N1B—H13B	111.7 (19)	C1A—C2A—H2A	120.73
H11B—N1B—H12B	106 (3)	C2A—C3A—H3A	117.33
C2—C1—C7	121.28 (16)	C1A^{i}—C3A—H3A	123.45
C2—C1—C6	118.74 (18)	N1B—C1B—C2B	119.32 (19)
C6—C1—C7	119.98 (19)	N1B—C1B—C3B^{ii}	119.07 (18)
C1—C2—C3	120.17 (17)	C2B—C1B—C3B^{ii}	121.62 (17)
O2—C2—C1	121.69 (18)	C1B—C2B—C3B	119.15 (19)
O2—C2—C3	118.14 (18)	C1B^{ii}—C3B—C2B	119.23 (19)
C2—C3—C4	120.25 (19)	C1B—C2B—H2B	120.57
C3—C4—C5	120.12 (19)	C3B—C2B—H2B	120.28
S5—C5—C4	120.31 (15)	C2B—C3B—H3B	125.22
S5—C5—C6	119.80 (16)	C1B^{ii}—C3B—H3B	115.25

O51—S5—C5—C4	−28.54 (18)	O2—C2—C3—C4	179.57 (18)
O51—S5—C5—C6	152.61 (15)	C2—C3—C4—C5	0.4 (3)
O52—S5—C5—C4	−148.51 (16)	C3—C4—C5—C6	−0.1 (3)
O52—S5—C5—C6	32.64 (18)	C3—C4—C5—S5	−178.92 (16)
O53—S5—C5—C4	91.59 (17)	S5—C5—C6—C1	178.78 (14)
O53—S5—C5—C6	−87.26 (17)	C4—C5—C6—C1	−0.1 (3)
C6—C1—C2—O2	−179.73 (18)	C3A^{i}—C1A—C2A—C3A	0.0 (3)
C6—C1—C2—C3	0.5 (3)	N1A—C1A—C2A—C3A	179.79 (19)
C2—C1—C6—C5	−0.1 (3)	C2A—C1A—C3A^{i}—C2A^{i}	0.0 (3)
C7—C1—C6—C5	179.55 (17)	N1A—C1A—C3A^{i}—C2A^{i}	−179.79 (18)
C7—C1—C2—C3	−179.18 (18)	C1A—C2A—C3A—C1A^{i}	0.0 (3)
C6—C1—C7—O72	169.88 (18)	N1B—C1B—C2B—C3B	−179.62 (19)
C2—C1—C7—O71	171.06 (19)	C2B—C1B—C3B^{ii}—C2B^{ii}	−0.2 (3)
C7—C1—C2—O2	0.6 (3)	C3B^{ii}—C1B—C2B—C3B	0.2 (3)
C2—C1—C7—O72	−10.4 (3)	N1B—C1B—C3B^{ii}—C2B^{ii}	179.62 (18)
C6—C1—C7—O71	−8.6 (3)	C1B—C2B—C3B—C1B^{ii}	−0.2 (3)
C1—C2—C3—C4	−0.7 (3)		

Symmetry codes: (i) −*x*, −*y*, −*z*+1; (ii) −*x*+1, −*y*+1, −*z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O72	0.88 (4)	1.74 (4)	2.558 (2)	152 (4)
N1*A*—H11*A*···O71^{iii}	0.99 (4)	2.41 (4)	3.114 (2)	128 (3)

N1*A*—H11*A*···O72^{iii}	0.99 (4)	1.84 (4)	2.808 (3)	168 (3)
N1*B*—H11*B*···O53	0.96 (3)	1.90 (3)	2.812 (2)	157 (3)
N1*A*—H12*A*···O71^{iv}	0.91 (3)	1.83 (3)	2.725 (3)	166 (3)
N1*B*—H12*B*···O52^{v}	0.89 (3)	2.01 (4)	2.868 (3)	161 (3)
N1*A*—H13*A*···O2^{vi}	0.90 (3)	2.43 (3)	3.015 (3)	123 (2)
N1*A*—H13*A*···O51^{i}	0.90 (3)	2.14 (3)	2.905 (3)	143 (3)
N1*B*—H13*B*···O51^{vii}	0.84 (3)	1.95 (3)	2.794 (3)	177 (3)
C2*A*—H2*A*···O72^{iii}	0.95	2.50	3.249 (2)	136
C3—H3···O71^{iv}	0.95	2.54	3.192 (3)	126
C4—H4···O53^{vii}	0.95	2.59	3.385 (3)	141

Symmetry codes: (i) −*x*, −*y*, −*z*+1; (iii) −*x*+1, −*y*+1, −*z*+1; (iv) *x*−1, *y*, *z*; (v) −*x*+1, −*y*, −*z*; (vi) −*x*, −*y*+1, −*z*+1; (vii) −*x*, −*y*, −*z*.